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EVALUATION OF CANDIDATE STIRLING ENGINE HEATER TUBE ALLOYS AT 820° AND 860° C

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ABSTRACT

Seven commercial alloys were evaluated in the NASA Lewis Research Center Stirling simulator materials rigs. Five iron-base alloys (N-155, A-286, Incoloy 800, 19-9DL, and 316 stainless steel), one nickel-base alloy (Inconel 718), and one cobalt-base alloy (HS-188) were tested in the form of thin-wall tubing in a diesel-fuel-fired test rig. Tubes filled with hydrogen or helium at gas pressure of 21.6 MPa and temperatures of 820° and 860° C were endurance tested for 1000 and 535 hours, respectively.

Results showed that under these conditions hydrogen permeated rapidly through the tube walls, thus requiring refilling during each 5-hour cycle. Helium was readily contained, exhibiting no measurable loss by permeation. Helium-filled tubes tested at 860° C all exhibited creep-rupture failures within the 535-hour endurance test. Subsequent tensile test evaluation after removal from the rig indicated reduced room temperature ductility for some hydrogen-filled tubes compared to helium-filled tubes, suggesting possible hydrogen embrittlement in these alloys.

SUMMARY

Seven tubing alloys were endurance tested in a diesel fuel fired, Stirling engine simulator material test rig. During exposure two sets of each alloy were pressurized - one with commercial purity hydrogen, and the other with helium. The maximum pressures ranged from 17.2 to 21.6 MPa. Two endurance runs were conducted, one at 860° C for 535 hours and the other at 820° C for 1000 hours. The alloys tested in the 860° C endurance run were N-155, A-286, Incoloy 800, 19-9DL, 316 stainless steel, and Inconel 718. For the endurance run at 820° C HS-188 was substituted for 316 stainless steel. All alloys were in the form of tubing with an outside diameter of 4.8 mm and a wall thickness of 0.8 mm. Hydrogen permeated rapidly through the walls of all alloys when heated to the operating temperature (860° or 820° C) and repressurization was required every 5 hours. In contrast, helium was readily contained. Creep-rupture failures occurred in all six alloys endurance tested at 860° C when pressurized with helium. Three alloys did not fail when pressurized with hydrogen - N-155, 19-9DL, and A-286. During the endurance test at 820° C for 1000 hours only one alloy, HS-188, did not fail when pressurized with helium. The alloy N-155 did not fail when pressurized with hydrogen but did have failures when pressurized with helium.

Rig exposure at 860° C resulted in subsequent decreases in room temperature strength for the 19-9DL alloy and for Inconel 718 when subsequently tested at 820° C. Ductility losses were noted for all alloys in subsequent

room temperature tests. The ductility loss was greater in the hydrogen-filled tubes in the Incoloy 800 indicating possible hydrogen embrittlement. Exposure at 820° C for 1000 hours resulted in subsequent reductions in room temperature strength of all alloys containing hydrogen. Lower room temperature ductility of hydrogen-filled tubes were noted for five of the six alloys. This decreased ductility was significant for N-155, Incoloy 800, 19-9DL, and Inconel 718 alloys.

INTRODUCTION

The work described in this report was conducted as part of the continuing supporting research and technology activities under the DOE-NASA Stirling Engine Highway Vehicle Systems Program.(1) To achieve maximum efficiency in the Stirling engine, hydrogen is used as the working fluid and operating temperatures are kept high, near 860° C in some proposed engine designs. In current prototype engines, N-155, an alloy containing 20 percent cobalt is used for the heater head tubing. Because of the strategic nature of cobalt (greater than 95 percent import dependence by the United States), its limited availability compared to the mass market needs for proposed automotive Stirling engine applications, and high-cost alloys containing cobalt cannot be considered for automotive applications.(2)

Planned automotive applications of the Stirling engine require cyclic on-off operation with resulting temperatures on the heater head tubes ranging from room temperature to near 900° C.(3) In addition, speed of the automobile is controlled by pressure variation of the hydrogen working fluid contained in the heater head tubes. Pressure normally ranges between 2 and 15 MPa with an average pressure near 7 MPa based on a 50 percent Urban/50 percent highway driving cycle.

The purpose of this investigation was to rank candidate alloys that may be substitutes for the cobalt containing alloy, N-155, used in current prototype engines. An accelerated test was conducted by using a pressure of about 21 MPa (1 MPa = ~143 psi) in the tubes. This resulted in a stress in the tubes that was much more severe then in actual engine operation but provided a means of ranking the alloys in a reasonable length of time.

Alloys evaluated included N-155 and six other heater tube alloys under conditions of cyclic temperature, pressure, and environment (hydrogen and combustion products) that simulate the actual operation of a Stirling-powered highway vehicle. The alloys tested were N-155 (the heater head tubing alloy currently used in prototype engines), four iron base candidate alloys, A-286, Incoloy 800, 19-9DL, and 316 stainless steel, a nickel-base alloy Inconel 718 and a cobalt-base alloy HS-188.

The iron base alloys and the nickel base alloy are considered possible candidates for the Stirling engine. The cobalt-base alloy, HS-188, was included in the program to determine the effects of the various test parameters (primarily hydrogen environment) on the three commonly used high-temperature iron, nickel, and cobalt-base alloys.

To date three endurance runs have been completed using the Stirling engine simulator test rigs. The first was an endurance run for 1000 hours at 760° C, the second was for 535 hours at 860° C and the third was for 1000 hours at

820° C. The results of the first endurance run, along with the details of the Stirling engine simulator rig configuration and operating procedure, have been reported previously (4). The results of the second and third endurance tests will be discussed in this report.

EXPERIMENTAL PROCEDURES

Materials

The alloys used in this study were obtained commercially in the form of tubing with an outside diameter of 4.8 mm and an inside diameter of 3.2 mm. Four of the alloys were weld drawn: N-155, 19-9DL, Inconel 718, and HS-188; and three were seamless: A-286, Incoloy 800 and 316 stainless steel. In the endurance test at 860° C the alloys used were: N-155, A-286, Incoloy 800, 19-9DL, 316 stainless steel, and Inconel 718. For the endurance run at 820° C the alloy HS-188 was substituted for 316 stainless steel. The remaining five alloys were the same in both endurance runs. Chemical analyses of the alloys as reported by the fabricator are shown in table I. One alloy is stainless steel (316). Four alloys (N-155, A-286, Incoloy 800 and 19-9DL) are iron-base super-alloys with substantial amounts of nickel and chromium. One alloy, Inconel 718 is a nickel base alloy and HS-188 is a cobalt base alloy. Average grain sizes ranged from 7-μm for HS-188 to 73-μm for A-286. Prior to the endurance runs, several of the alloys were heat treated to increase the grain size and thus, improve the elevated temperature creep-rupture properties. The alloy heat treatments and grains sizes are as follows:

Alloy	Solution treatment	Atmosphere	Time	Cooling	Grain size, μm
N-155	1204° C	Vacuum	10 min	Fce Cool	17
A-286	1148° C	Vacuum	10 min	Fce Cool	73
Incoloy 800	1121° C	Vacuum	1 hour	Fce Cool	66
19-9DL	1204° C	Vacuum	10 min	Fce Cool	25
316 Stainless Steel	1093° C	Vacuum	3 min	Helium Quench	125

The microstructures of all alloys before the endurance runs are shown in figure 1.

The tubing alloys were evaluated in the Stirling engine materials test rig in the form of a "hairpin" as shown in figure 2. Each leg of the "hairpin" was 30.5 cm long with 2.5 cm between legs. Four of these "hairpin" tubes were attached to a copper header with internal passages and external tubes and valves for filling the four "hairpins". The copper header and attached tubes comprised a module. A module is shown in figure 3. The "hairpins" were attached to the copper header with gas-tight mechanical connectors. Also shown in figure 3 is the thermocouple that was attached at the bottom of one of the "hairpin" tubes as well as the lead to the pressure transducer that was located adjacent to the thermocouple plug at the top of the module. The "hairpin" tubes were connected in series and were filled through the tubing and valves located on top of each copper header. When one tube failed, the pressure in the remaining three tubes was lost.

Before installation in the rig each assembled module was leak checked to 10 MPa with helium by submersion in water. The modules were previously proof tested to 30 MPa by pressurizing with an organic Stratton solvent (Varsol). Little difficulty was experienced in achieving leak proof modules.

Analyses of the commercial grade hydrogen used in the endurance run at 860° C are shown in table II, the hydrogen analyses for the endurance run at 820° C are shown in table III. Note that the oxygen contents of the various hydrogen bottles used in the endurance run at 860° C varied from zero to 1648 ppm and is representative of commercial purity hydrogen, while that used in the endurance run at 820° C varied from zero to 366 ppm and is representative of commercial high-purity hydrogen. A large number of hydrogen bottles were required due to the rapid permeation of hydrogen through the tube walls when at temperature. This required the refilling of the modules every five hours during each endurance run. Analyses of the helium used revealed no detectable amounts of oxygen.

Stirling Engine Simulator Rig

The Stirling engine simulator rig used in this program was designed and fabricated at the Lewis Research Center; it consists primarily of a combustion gas heating chamber with auxilary heating, control and gas management systems. A schematic of the heating chamber is shown in figure 4. A detailed description of the rig and its operation are given in reference 4.

An endurance run consisted of a series of 5-hour cycles to obtain the required exposure time, 535 hours at 860° C and 1000 hours at 820° C. A typical heating cycle was made up of a six to ten minute preheat to get to the operating temperatures, a 5-hour hold at temperature, followed by a cool down to near room temperature.

The cool down time between cycles in the furnace was 1 hour or longer. After cooldown to about 25° C the modules containing hydrogen were vented, pressure transducers rezeroed and refilled. The modules containing helium did not require refilling after each cycle: they were evacuated and refilled only after "hairpin" replacement following failure or leak. The helium pressure was maintained at 17 to 21 MPa during the 5-hour cycles until two "hairpins" of the module failed. After this, the pressure in that module was reduced to 1 to 2 MPa to prevent further hairpin failures and thus permit post-test mechanical property comparisons with hydrogen-containing tubes that generally lasted longer. After completion of the test run or after failure, the "hairpins" were removed from the modules and sectioned for tensile testing and metallographic examination.

The temperature profile inside the heating chamber of the rig was determined at the start of each endurance run. The profile was determined by measuring the temperature at three locations on each module. The thermocouples were fastened to each module at the bottom bend of the "hairpin" as well as at 10.2 cm and at 22.9 cm from the bottom bend.

The modules were modified at the end of the 860° C endurance run to eliminate a "hot-zone" at the top of the "hairpin", approximately 23 cm from the bottom bend. This modification consisted of the installation of a 10.2 cm long sleeve (9.5 mm diameter) around each tube leg in the "hot zone". This

effectively reduced the temperature variation along each "hairpins" of each module.

The temperature profile determined for the two endurance runs are shown in figures 5(a) and (b). This figure shows the temperature at the top, middle and bottom locations on the module plotted against angular position around the furnace. Also shown are the alloys at the module location. Figure 5(a) shows the results for the 860° C endurance run. The temperatures at the top varied from 840° to 882° C with the highest temperature at the 60 degree (module 2) location. This is the location of the Inconel 718 alloy. The maximum temperature at the 270 degree location (module 9) was only 840° C. This is the location of the A-286 alloy (hydrogen module). The temperatures at the middle and bottom locations were substantially lower than those at the top location and varied from 808° to 821° C at the bottom locations and 810° to 823° C at the middle locations. Most tubing failures occurred in the top locations of the "hairpins". Figure 5(b) shows the temperature profile for the 820° C endurance run in which the 10.2 cm sleeves were attached to the module at the top location. The temperatures at the top varied from 798° to 838° C with the highest temperature again at the 60 degree (module 2) location. The temperatures at the middle location varied from 798° to 808° C. At the bottom location the temperatures varied from 802° to 825° C. Comparison of figures 5(a) and (b) shows that the 10.2-cm sleeves at the top location (figure 5(b)) reduced the temperatures at this location, thus reducing rig temperature variation.

Post Exposure Evaluations

Post exposure evaluation included both tensile testing at room temperature and 820° C and metallography.

Tensile tests were conducted on tube specimens at room temperature and at 820° C. The tensile grips and a tensile specimen are shown in figure 6. The bottom of the tensile specimen is enclosed in the grips. An exploded view of the grip components is shown at the top of the specimen. The tensile specimens were 10.2 cm long when tested at room temperature and 17.8 cm long when tested at 820° C. A solid tool steel pin 2.5 cm long was inserted into the tube ends to prevent collapse of the tube during testing. The elongation after testing was determined from measurements of prescribed marks on the tensile specimens. All tests were conducted at a crosshead speed of 0.25 cm/min.

Metallographic specimens were sectioned from the "hairpin" tubes taken from each module. The specimens were polished, appropriately etched and examined at magnifications of 100X and 500X.

RESULTS

Microstructure

As previously indicated microstructures of the alloys before the endurance runs are shown in figures 1(a) through (g). Typical microstructures after endurance testing at 820° C are shown in figure 7(a) and (b). This is for the 19-9DL alloy exposed to hydrogen pressure. Minor changes in microstructure were observed in the 19-9DL alloy. These included growth and coalescence of

the carbide particles in the matrix and grain boundaries. Figure 7(b) also shows that the wall thickness of the 19-9DL alloy was severely reduced after 1000 hours of the endurance test. Measurements of tubing outside diameter show that approximately 50 percent of the tube wall was lost, indicating that the fireside burner environment had caused oxidation and spalling of the tubing outside surface. Note that there is little retained oxide on the outside surface of the tube, figure 7(a). Photomicrographs of the outside edge of two other alloys after endurance testing are shown in figures 8(a) and (b), A-286 and Incoloy 800 respectively. Note the presence of adherent scale on the A-286 shown in figure 8(a) and the grain boundary oxidation in the Incoloy 800, figure 8(b). Weight-loss measurements made on oxidation specimens attached at the bottom of the "hairpins" also showed that the 19-9DL alloy had weight losses of 30 to 50 percent in the burner environment. None of the other alloys exhibited this behavior. No weight losses were found on any of the other tube "hairpins" during the 1000 hour endurance run at 820° C.

Hydrogen Permeability

860° C – 535 hours

Hydrogen pressure-time data for the six alloys are presented in table IV. The data are from six selected cycles during the 535 hour endurance test at 860° C. The six cycles selected were those for 88, 134, 236, 373, 414 and 519 hours during the run. The data show a rapid rise in pressure to 17 to 22 MPa, usually within 10 minutes, due to increasing temperature and then a decay in pressure during the remainder of the cycle. This pressure decay with time at temperature results from permeation of hydrogen through the hot walls of the tube and varied with oxygen content of the hydrogen and with time during the endurance run. The data show large reductions in pressure decay occurred in N-155, 316 stainless and Inconel 718. Smaller changes took place in A-286, Incoloy 800 and 19-9DL. Typical pressure decay curves for Incoloy 800 and Inconel 718 are shown in figures 9 and 10, respectively. Note that in figure 10 for Inconel 718, the pressure decay curves show a larger variation of pressure decay with time during the 535 hour endurance run, whereas there was little change in pressure decay characteristics for the Incoloy 800 alloy as shown in figure 9.

Using the equation:

$$P^{1/2} = P_o^{1/2} - \varphi \frac{A P_s T_1 t_1}{2\ell V_1 T_2} \qquad \text{(ref. 4)}$$

the permeability coefficient 0 was calculated for each alloy, for selected 5-hour cycles throughout the 535 hour endurance run. The results are shown in table V. The results show that the permeability coefficient was not constant for all alloys nor was it constant for a given alloy throughout the test. This was also the case in the endurance run at 760 discussed in reference 2. In both endurance runs (760° and 860° C) a correlation between oxygen + CO_2 content of the hydrogen and the resulting permeability coefficient exists for some alloys. The higher the oxygen + CO_2 content of the hydrogen, the lower the resulting permeability coefficient. This is particularly true for the N-155 and Inconel 718 alloys. Data for Inconel 718 are shown in figure 11.

820° C - 1000 hours

Hydrogen pressure-time data for the six alloys are presented in table VI. The data are from five selected 5-hour cycles of the 1000 hour endurance test at 820° C. The 5-hour cycles selected were those for 95, 296, 500, 754 and 996 hours of the endurance test. No data are shown for a given alloy after the second tube failure of that alloy in a given module.

The calculated permeability coefficients for the 820° C endurance run are given in table VII. The results show that the permeability coefficient varied from 2.2 X 10^{-5} $cm^2/sec\ MPa^{1/2}$ for A-286 after 5 hours to 2.4 X ~ 10^{-6} $cm^2/sec\ MPa^{1/2}$ for Inconel 718 after 250 hours. There was a substantial decrease in permeability coefficient during the first 105 hours of the endurance run for all alloys except 19-9DL, where little change in permeability coefficient was found. The reductions in permeability coefficient varied from 44.7 percent for HS-188 to 70 percent for Inconel 718. The 19-9DL alloy showed only a 0.05 percent reduction during the first 100 hours into the endurance test.

Examination of the analyses of hydrogen used throughout the 1000 hour endurance run, table III, shows that the oxygen content of all hydrogen used, averaged 167 ppm with a variation from zero to 326 ppm and the CO_2 content varied from 0 to 1405 ppm. A factor considered to have a large influence on apparent permeability rates is the conditions of the surface of the metal, references 4 and 5. In particular, the presence or absence of any surface oxide should have a large influence on the apparent permeability of hydrogen through the tube wall. Any oxide films on the inside or outside surface of the tube will reduce the rate of hydrogen permeation through the wall. Examination of tables II and III which give the oxygen and CO_2 contents of the hydrogen used during both endurance runs and tables VI and VII which give the permeability coefficients show that reduced hydrogen permeability for some alloys (e.g., INCONEL 718 - fig. 11) can be correlated with the introduction of a high oxygen + CO_2 content hydrogen bottle at a particular time of test. This effect was reversible with the introduction of hydrogen with lower oxygen content. In general the higher the oxygen content of the hydrogen the lower was the hydrogen permeability.

As noted previously under microstructure, a thick oxide layer was found on the fire-side surface of the A-286 alloy and lesser amounts on the remaining alloys, except 19-9DL where little or no oxide was present. Metallographic examination also showed no measureable oxide layers on the inside surfaces of the tubes. It is postulated that the presence of an oxide layer on the fireside of the tube in conjunction with a thin (sub-microscopic) oxide layer on the inside are dominant factors in the reduction of hydrogen permeation through the tube walls at the operating temperatures (ref. 5).

Tubing Failures

Many of the "hair-pin" tubes failed during the two endurance tests. The tubes that contained helium had more failures than did those that contained

hydrogen due to the constant higher pressure maintained in the helium-containing tubes throughout each of the 5-hour cycles. In contrast the hydrogen pressure generally decayed with time and thereby reduced the stress in the tube wall for part of each 5-hour cycle. The tube failure times are shown in tables VIII(a) and (b).

860° C - 535 hours

The failure times for the tubes during the 860° C endurance run are shown in table VIII(a). Three of the six alloys tested N-155, A-286 and 19-9DL had no failures in the tubes which contained hydrogen; however, failures occurred in all tubes which contained helium. The Inconel 718 alloy tubes had the earliest failures of those tubes which contained hydrogen. These early failures of the Inconel 718 alloy tubes which contained hydrogen indicate a possible hydrogen degradation in this alloy. Alternatively the retention of hydrogen because of low permeability may have caused the early failures due to a higher stress in the tubes. The lower strength Incoloy 800 and the 316 stainless steel alloy tubes had the earliest failure times at the 860° C operating temperature when pressurized with helium.

820° C - 1000 hours

The failure times for the tubes during the 820° C endurance run are shown in table VIII(b). Hydrogen-filled N-155 and HS-188 did not exhibit failures. Helium-filled HS-188 did not fail. The lower strength alloys A-286 and Incoloy 800 had the earliest failures when pressurized with helium (31 hours and 150 hours, respectively). Incoloy 800 also had the earliest failure time when pressurized with hydrogen (145 hours).

Comparison of the failure times for a given alloy in tables VIII(a) and VIII(b) show that for tubes which contained helium there were minor differences in failure times for most alloys. Most alloys, as expected, survived slightly longer at the lower (820° C) operating temperature, however, the A-286 alloy survived longer at the 860° C operating temperature than at 820° C when pressurized with helium.

Tensile Properties

The tensile properties of the as-received and/or heat treated tubing were determined. After completion of the endurance runs, the "hairpin" tubes of all the alloys were sectioned for tensile testing to determine the effect of rig environment on the tensile properties. Each of the alloys had been exposed to both hydrogen and helium in addition to the burner combustion products. Both room-temperature and high temperature (820° C) tensile tests were conducted. Two specimens of each alloy were tested at room temperature, and at least one specimen representative of each alloy and environmental exposure condition was tested at 820° C. The results of these tests are described in the following sections.

As-received properties. - The room temperature and 820° C tensile properties of the tubing alloys both prior to the endurance runs as well as after exposure are presented in tables IX(a) and (b) for the 860° C run and in tables X(a) and (b) for the 820° C run. The alloys used in the two endurance

runs were identical except that HS-188 was substituted for 316 stainless steel in the endurance run at 820° C.

860° C - 535 Hours

The room temperature tensile properties of the six alloys used in the 860° C endurance run are given in table IX(a). The ultimate strengths ranged from a low of 552 MPa for Incoloy 800 to a high of 878 MPa for N-155. The tensile elongation for all six alloys were good and ranged from 39 percent for Incoloy 800 to 60.3 percent for 19-9DL. The elevated temperature strength at 820° C of the six alloys are shown in table IX(b). The strength at 820° C ranged from 160 MPa for 316 stainless steel to 519 MPa for Inconel 718. The tensile elongation at 820° C ranged from 19.0 percent for Inconel 718 to 62.0 percent for the lower strength Incoloy 800.

820° C - 1000 Hours

The room temperature ultimate strengths of the six alloys used in the 820° C endurance run are given in table X(a). The strengths ranged from a low of 552 MPa for Incoloy 800 to a high of 1091 MPa for HS-188. The tensile elongation for all six alloys were good and ranged from 35.0 percent for HS-188 to 60.3 percent for 19-9DL. The elevated temperature strength at 820° C of the six alloys are shown in table X(b). The strength at 820° C ranged from 162 MPa for Incoloy 800 to 519 MPa for Inconel 718. The tensile elongation at 820° C ranged from 19.0 percent for Inconel 718 to 62.0 percent for Inconel 800.

860° C - 535 Hours

Post-endurance test properties. - The room temperature tensile data for the six alloys prior to and after exposure at 860° C, while under pressure with helium and hydrogen for 535 hours, are given in table IX(a) and shown graphically in figure 12. Exposure in the rig resulted in little or no change in room temperature strength for four of the six alloys. One alloy, 19-9DL, showed a reduction in ultimate strength from 767 MPa to 604 MPa for those tubes which contained helium and to 611 MPa for those tubes which contained hydrogen. One alloy, Inconel 718, had higher strength after exposure than before. Except for Incoloy 800, five of the six alloys exhibited similar reductions in ductility (percent elongation) for both hydrogen and helium-filled tubes. Reduction in ductility was substantial for all five alloys. However, for the Incoloy 800 alloy, the tubes which contained helium showed minimal loss in ductility while tubes containing hydrogen suffered a 25 percent loss in ductility indicating possible hydrogen embrittlement in this alloy.

The elevated temperature (820° C) tensile data for the six alloys prior to and after exposure at 860° C are given in table IX(b) and are shown in figure 13. Exposure in the rig for 535 hours resulted in a large decrease in tensile strength for the Inconel 718 alloy. The ductility however was not degraded, the remaining five alloys exhibited very little change in strength after rig exposure and there was no consistent behavior in change in ductility with helium or hydrogen environment.

820° C - 1000 Hours

The room temperature tensile data for the six alloys prior to and after exposure at 820° C for 1000 hours are given in table X(a) and are shown in figure 14. Exposure in the rig resulted in some loss in room temperature strengths for all six alloys when pressurized with hydrogen while helium-filled tubes experienced an increase in strength for four of the six alloys. The loss in strength for hydrogen-filled tubes was most pronounced in the N-155, 19-9DL and Inconel 718 alloys. For N-155 the loss in strength was from 878 MPa to 734 MPa (16 percent) after exposure. For 19-9DL the loss was from 767 to 655 MPa (15 percent). For Inconel 718 the loss was from 875 MPa to 772 MPa (12 percent). For these three alloys the loss in room temperature tensile strength was accompanied by a substantial decrease in ductility as measured by percent elongation. These losses were as follows: N-155 from 45 percent to 8.5 percent, 19-9DL from 60.3 percent to 7 percent, and Inconel 718, from 41.5 percent to 9 percent. These large losses in ductility accompanying the losses in ultimate tensile strength indicates possible hydrogen embrittlement for the three alloys N-155, Inconel 718 and 19-9DL. There is also possible embrittlement of the Incoloy 800 alloy as there was a slight loss in strength (552 MPa to 538 MPa) accompanied by a substantial loss in ductility as measured by percent elongation (from 39 percent to 25 percent). For the above four alloys the loss in ductility of the tubes that contained hydrogen was much greater than in the tubes which contained helium.

The elevated temperature (820° C) tensile data for the six alloys after exposure at 820° C for 1000 hours are given in table X(b) and are shown plotted in figure 15. Exposure in the rig with hydrogen in the tubes resulted in a large decrease in tensile strength at 820° C for the Incoloy 718 alloy (519 MPa to 361 MPa). Smaller losses occurred in the N-155 alloy (296 MPa to 238 MPa) and the HS-188 alloy (397 MPa to 367 MPa). There was also some reduction in ductility for three of the six alloys tested, Incoloy 800, 19-9DL and HS-188, which contained hydrogen.

DISCUSSION

The results of the two endurance runs again show that helium can readily be contained in the heater tubes, at a pressure of 21.6 MPa and at temperatures of 860° and 820° C, however, hydrogen permeates through the tube walls and must be replenished periodically. The rate of hydrogen permeation was found not to be constant but rather is variable and is influenced by alloy composition and oxygen + CO_2 content of the hydrogen used. The reduction of hydrogen permeability during the endurance tests is thought to be associated with the formation of an oxide layer on the fire-side of the tube coupled with the formation of a very thin oxide film on the inside surface of the tube. Table XI, which summarizes the findings of this report, shows that alloys A-286, Incoloy 800, 19-9DL, and 316 stainless steel formed an oxide layer on the fire-side of the tube. However, this external oxide on the fireside of the 19-9DL tubes spalled severely thus, reducing the tube wall thickness. The 19-9DL alloy had the highest hydrogen permeability of all alloys. The lack of tenacity of the fire-side oxide of the 19-9DL alloy may be a significant factor in the rapid permeation of hydrogen through this alloy. Losses in wall thickness of 60 percent were found after 535 hours. No such losses in wall thicknesses were noted in the other alloys, where the fire-side oxide layers were more adherent.

Several alloys showed large reductions in hydrogen permeability when the hydrogen contained more than 350 ppm of oxygen + CO_2. These were N-155, 316 stainless steel and Inconel 718. Even though N-155 and Inconel 718 did not form thick oxide layers on the fire-side of the tube, the oxygen + CO_2 contained in the hydrogen did result in a significant reduction in hydrogen permeability. Thus, it is postulated that a thin oxide layer on the inside of the tubes is a dominant factor in the reduction of hydrogen permeation.

Sheet specimens of the alloys in the as-received condition have been evaluated in creep-rupture in another part of the Stirling materials evaluation program (ref. 6). These referenced results are shown in table XII and XIII. Based on a hoop stress in the tube of 50 MPa, the rupture lives predicted from the creep-rupture study varied from 8 hours for 316 stainless steel to 1411 hours for N-155, at the 860° C operating temperature. During the endurance run at 860° C, the average lives of the helium-filled tubes varied from 43 hours for Incoloy 800 to 511 hours for N-155, table XII. In general there is an agreement between predicted and actual ratings of the alloys. The rupture lives of the alloys A-286 and Inconel 718 are reversed as are those for 316 stainless steel and Incoloy 800, however the differences in rutpure lives are not large. For the endurance run at 820° C the average failure lives, varied from 31 hours for the Incoloy 800 alloy to no failures for 1000 hour for the HS-188 alloy, table XIII. The predicted rupture lives at 820° C varied from 176 hours for Incoloy 800 to 50,000 hours for HS-188. The rating of the alloys predicted from creep-rupture data agrees exactly with the rating from the tubing failures. Lack of agreement between predicted data from creep testing and actual rig results may be attributed in part to the cyclic nature of the rig testing, temperature gradients in the tubes, and the hostile environment from the diesel fuel-tired burner.

It was concluded from the first endurance run (ref. 4) that the small grain size (9-μm) of the 19-9DL alloy used in the first run was a major factor in the early failures of that alloy. The larger grain size of the tubing used in these two endurance runs (25-μm) did increase the failure time in both the 860° and 820° C endurance runs for 19-9DL. The rapid loss of fire-side oxide and thus wall thickness prevented attainment of the four-fold increase in rupture life as predicted from the creep-rupture data.

A comparison was made of the tensile properties after rig exposure at 860° C for 535 hours and at 820° C for 1000 hours. The comparison suggested that only the iron base alloy, Incoloy 800 may have experienced hydrogen embrittlement based on loss in room temperature ductility for hydrogen-filled tubes and helium-filled tubes after both rig exposures. In addition the nickel base alloy, Inconel 718 experienced a substantial loss in 820° C tensile strength after both rig exposures. The loss was comparable for helium- and hydrogen-filled tubes suggesting an aging effect rather than hydrogen degradation. Other than the aforementioned results, no notable difference was apparent for the iron, nickel, and cobalt base alloys in regard to their behavior in the hydrogen environment.

SUMMARY OF RESULTS

Endurance testing of seven tubing alloys in a diesel fuel-fired Stirling engine simulator materials test rig at 860° and 820° C and 17.0 to 21.6 MPa pressure with hydrogen and helium had the following results:

1. While helium was readily contained in all alloys, all alloys lost hydrogen rapidly at both temperatures, during the 5-hour segments of the endurance runs when pure hydrogen was used. Hydrogen loss rates for N-155, 316 stainless steel, and Inconel 718 decreased significantly when the oxygen + CO_2 content of the hydrogen was above 350 ppm. However, the high loss rates reoccurred in subsequent cycles when pure hydrogen (less than 350 ppm oxygen + CO_2) was used.

2. The observed reduced hydrogen permeabilities are believed to be associated with the formation on an adherent oxide on the fire-side of the tube coupled with the formation of a thin oxide film on the inside surface of the tube.

3. <u>860° C</u>

a. At 860° C all of the alloys when pressurized with helium at 20 MPa, had at least two tubing failures within 535 hours of exposure in the rig.

b. Rig exposure for 535 hours at 860° C did not seriously degrade the tensile strength of five of the alloys in subsequent tests at room temperature and 820° C. Strength losses were noted for 19-9DL when tested at room temperature and for 19-9DL, Incoloy 800, and Inconel 718 when tested at 820° C. Ductility losses were noted for all alloys when tested at room temperature after exposure. Generally the losses were similar for hydrogen- and helium-filled tubes except in Incoloy 800 where a larger loss in ductility was noted for the hydrogen-filled tubes indicating possible hydrogen embrittlement in this alloy.

4. <u>820° C</u>

a. At 820° C the HS-188 alloy had the longest life at the 20 MPa pressure. No tube failures occurred when pressurized with either helium or hydrogen during the 1000 hour endurance run. All other alloys had one or more tube failures when pressurized with helium.

b. Rig exposure for 1000 hours at 820° C did degrade the subsequent room temperature tensile strength of all alloys when pressurized with hydrogen. Larger reductions in room temperature ductility for hydrogen-filled tubes were noted for five of the six alloys. This was significant for the Incoloy 800 and 19-9DL.

c. The wall thickness of the 19-9DL alloy was reduced 60 percent during the endurance test at 820° C. The reduced wall thickness was the result of rapid metal consumption due to oxide spalling from the fire-side surfaces. All remaining alloys developed tight adherent oxides on the fire-side surfaces.

CONCLUDING REMARKS

Based on failure time in the Stirling engine materials simulator rig the candidate substitute alloys can be ranked as following in decreasing order of time to failure: 19-9DL, Inconel 718, A-286, 316SS, and Incoloy 800. Because of the severe oxidation spalling of 19-9DL, it would probably not be suitable for the 3500 hour life required for the automotive Stirling engine. Actual operating conditions (temperature, pressure, etc) will have to be clearly defined before it can be determined whether the remaining four alloys will be suitable for engine application based on their strength limitations in the accelerated testing reported herein.

Hydrogen embrittlement, although possibly present in some of the alloys, does not appear to be a serious problem since sufficient ductility remains in the tested tube specimens. Hydrogen permeability as indicated in our prior work continues to be a more serious problem, and means of reducing the permeability as well as identifying other candidate alloys must continue to assure Stirling Engine viability.

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TABLE I. - COMPOSITION, HEAT NUMBER, METHOD OF FABRICATION AND GRAIN SIZE FOR TUBING ALLOYS

Alloy Heat number Fabrication Grain size, μm	N-155 C102770 Weld drawn 17	A-286 27771 Seamless 73	Incoloy 800 HH6127A Seamless 66	19-9DL C35039 Weld drawn 25	316SS 07475 Seamless 25	Inconel 718 21B6ES Weld drawn 26	HS 188 1880-7-1579 Weld drawn 7
Element	Composition, weight percent[a]						
Chromium	21.2	14.4	22.5	18.3	17.3	17.7	22.0
Nickel	19.9	24.7	32.6	8.76	13.1	53.3	23.1
Cobalt	19.0	----	----	----	----	.03	Bal
Manganese	1.45	1.01	.73	1.02	1.57	.05	.67
Molybdenum	3.03	1.17	----	1.26	2.26	3.02	-----
Tungsten	2.61	----	----	1.18	----	----	13.9
Titanium	----	2.12	.52	.27	----	1.12	-----
Columbium	1.05	----	----	.37[b]	----	5.18[b]	-----
Silicon	.55	.62	.53	.47	.54	.20	.38
Aluminum	----	----	.54	----	----	.56	-----
Carbon	.11	.06	.01	.29	.06	.04	.11
Nitrogen	.16	----	----	----	----	----	-----
Copper	----	.32	.03	----	----	.04	-----
Vanadium	----	.26	----	----	----	----	-----
Boron	----	----	----	----	----	----	.003
Lanthanum	----	----	----	----	----	----	.03
Iron	Bal	Bal	Bal	Bal	Bal	18.8	1.79

[a]Vendor analyses
[b]Cb/Ta

TABLE II. - HYDROGEN ANALYSES FOR COMMERCIAL GRADE HYDROGEN USED IN ENDURANCE RUN AT 860° C

Test time, hr	Analysis, ppm*					
	H_2O	N	O_2	Ar	CO_2	CH_4
0 to 37	----	2793	502	13	143	157
37 to 87	22.8	923	463	0	674	---
87 to 139	----	5000	0	0	157	121
139 to 198	----	1691	382	24	29	114
198 to 251	26.8	923	463	0	674	---
251 to 323	----	5177	62	11	196	---
323 to 403	----	4678	43	23	128	---
403 to 453	----	6252	1648	70	477	---
453 to 499	----	2910	0	0	240	---
499 to 535	----	2752	65	0	241	---

* ppm = parts per million.

TABLE III. - HYDROGEN ANALYSES FOR COMMERCIAL GRADE HYDROGEN USED IN ENDURANCE RUN AT 820° C

Test time, hr	Analysis, ppm*				
	N_2	O_2	Ar	CO_2	CH_4
0 to 30	2879	108	0	312	——
30 to 75	410	79	——	——	——
75 to 125	1673	87	0	1405	——
125 to 166	873	88	0	347	——
166 to 227	1046	326	34	28	——
227 to 296	790	20	42	——	——
296 to 356	834	207	0	0	156
356 to 405	1483	181	23	0	150
405 to 455	1196	164	0	29	116
455 to 500	1306	200	22	27	165
500 to 545	979	186	0	0	156
545 to 597	1584	220	44	84	116
597 to 664	1428	247	46	29	102
664 to 749	1514	260	23	——	165
749 to 809	2195	300	22	27	149
809 to 879	1647	319	45	28	132
879 to 955	149	0	0	0	0
955 to 1000	960	18	0	0	0

*ppm = parts per million.

TABLE IV. - HYDROGEN PRESSURE DECAY DURING SIX SELECTED CYCLES IN THE 535 HOUR ENDURANCE TEST AT 860° C

(a) N-155

Endurance test, hr											
88		134		236		373		414		519	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
9	18.1	8	19.3	7	19.3	9	19.4	11	21.1	9	20.1
15	17.2	21	17.2	----	----	31	17.2	----	----	----	----
60	13.9	60	13.4	60	17.9	60	15.3	60	18.8	60	19.2
120	10.5	120	9.1	120	16.4	120	11.2	120	17.4	120	18.2
180	7.8	180	5.8	180	15.0	180	8.3	180	16.3	180	17.5
240	5.6	240	3.9	240	13.8	240	5.9	240	----	240	16.8
300	4.0	300	2.1	300	12.6	300	4.0	300	14.3	300	16.1

(b) A-286

Endurance test, hr											
88		134		236		373		414		519	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
8	17.7	8	19.4	7	19.1	9	19.4	9	20.6	9	20.1
10	17.2	32	17.2	20	17.2	28	17.2	41	17.2	59	17.3
60	12.4	60	15.0	60	13.2	60	14.3	60	15.6	60	17.2
120	8.3	120	11.3	120	8.8	120	9.8	120	11.5	120	14.3
180	5.4	180	8.5	180	5.8	180	6.6	180	8.5	180	12.0
240	3.7	240	6.3	240	3.9	240	4.3	240	6.3	240	10.0
300	2.6	300	4.7	300	2.6	300	2.9	300	4.6	300	8.3

TABLE IV. - CONCLUDED

(e) 316 Stainless Steel

Endurance test, hr											
88		134		236		373		414		519	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
9	20.3	7	19.0	11	20.2	9	19.4	8	20.4	7	20.7
18	17.2	16	17.2	59	17.2	19	17.2	22	17.2	32	17.2
60	9.2	60	7.9	60	17.1	60	12.4	60	11.5	60	14.6
120	3.1	120	2.3	120	14.1	120	7.1	120	5.4	120	9.9
180	1.2	180	.5	180	11.6	180	3.6	180	2.3	180	6.8
240	.7	240	.3	240	9.5	240	1.5	240	1.0	240	4.3
300	.6	300	.3	300	7.8	300	.4	300	.6	300	2.6

(f) Inconel 718

Endurance test, hr											
88		134		236		373		414		519	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
12	20.0	8	19.4	8	20.0	9	19.4	15	21.6	12	20.6
—	—	49	17.2	—	—	29	17.2	—	—	—	—
60	19.4	60	16.0	60	19.6	60	13.6	60	21.1	60	19.6
120	19.1	120	12.1	120	18.8	120	9.0	120	20.3	120	18.2
180	18.6	180	9.2	180	17.9	180	5.9	180	19.5	180	16.8
240	18.1	240	6.8	240	17.1	240	3.7	240	18.8	240	15.3
300	17.6	300	4.6	300	16.2	300	2.3	300	18.0	300	13.8

TABLE IV. - CONTINUED

(c) Incoloy 800

Endurance test, hr											
88		134		236		373		414		519	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
9	18.6	8	19.4	10	19.9	9	19.8	10	21.0	9	19.7
32	17.2	26	17.2	32	17.2	19	17.23	31	17.2	26	17.2
60	15.4	60	13.8	60	14.3	60	13.1	60	13.4	60	13.6
120	11.9	120	9.2	120	9.8	120	6.9	120	8.1	120	8.6
180	9.2	180	6.1	180	6.7	180	3.5	180	4.7	180	5.3
240	7.0	240	3.9	240	4.5	240	2.0	240	2.3	240	3.3
300	5.3	300	2.6	300	3.0	300	1.03	300	1.7	300	1.7

(d) 19-9 DL

Endurance test, hr											
88		134		236		373		414		519	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
7	17.2	8	18.6	7	18.3	7	18.8	7	19.9	7	18.8
---	----	---	----	---	----	---	----	---	----	---	----
60	6.0	60	6.1	60	5.1	60	5.2	60	6.4	60	3.4
120	.9	120	.7	120	0.3	120	.3	120	1.0	120	.6
180	.2	180	.1	180	----	180	.1	180	.5	180	.2
240	.1	240	.1	240	----	240	.1	240	.4	240	.1
300	.1	300	.1	300	----	300	.1	300	.4	300	.1

TABLE V. - PERMEABILITY COEFFICIENTS FOR ENDURANCE TEST AT 860° C

Test time, hr	Oxygen content, ppm	Test time for calculated φ, hr	Permeability coefficient $\varphi \times 10^{-6}$ $cm^2/sec\ MPa^{1/2}$					
			N-155	A-286	Incoloy 800	19-9 DL	316 SS	Inconel 718
0-87	463/502	5	12.2	22.9	8.3	20.5	18.6	0.93*
0-87	463/502	87	3.4	4.8	3.1	9.4	8.9	.37
88-139	0	139	4.8	3.6	4.5	12.1	10.4	3.50
140-251	382/463	208	1.4	4.9	4.8	17.0	2.2	.26
252-403	43/62	383	3.6	4.4	6.2	14.4	5.6	4.80
404-453	1648	453	.45	3.0	3.3	12.5	7.1	.19
454-535	0/65	504	.96	2.4	5.3	13.9	5.0	.77

*22 hrs.

TABLE VI. - HYDROGEN PRESSURE DECAY DURING SIX SELECTED CYCLES IN THE 1000 HOUR ENDURANCE TEST AT 820° C

(a) N-155

Endurance test, hr									
95		296		500		754		996	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
12	20.3	12	18.6	12	19.6	12	19.4	13	20.2
27	17.2	20	17.2	30	17.2	29	17.2	41	17.2
60	11.6	60	11.9	60	13.0	60	13.2	60	15.4
120	5.2	120	7.6	120	8.6	120	8.9	120	10.7
180	1.9	180	4.6	180	5.3	180	5.6	180	7.2
240	.2	240	2.8	240	3.2	240	3.4	240	4.6
300	----	300	1.6	300	1.9	300	1.9	300	2.8

(b) A-286

Endurance test, hr			
95		296	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
12	20.3	12	17.7
29	17.2	16	17.2
60	11.1	60	9.5
120	5.6	120	4.6
180	2.8	180	2.2
240	1.2	240	.8
300	.3	300	.2

TABLE VI. - CONTINUED

(c) Incoloy 800

Endurance test, hr	
95	
Cycle time, min	Pressure, MPa
12	20.1
31	17.2
60	11.9
120	6.6
180	3.5
240	1.9
300	.9

(d) 19-9 DL

Endurance test, hr			
95		296	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
12	20.1	13	17.8
21	17.2	15	17.2
60	4.8	60	3.1
120	.4	120	.3
180	.3	180	.2
240	.3	240	.2
300	.3	300	.2

TABLE VI. - CONCLUDED

(e) HS-188

Endurance test, hr									
95		296		500		754		996	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
12	20.3	13	18.6	15	19.1	13	18.6	14	21.2
34	17.2	26	17.2	30	17.2	31	17.2	52	17.2
60	13.0	60	13.6	60	14.0	60	14.3	60	16.6
120	8.1	120	10.1	120	10.8	120	10.6	120	12.3
180	4.2	180	7.5	180	7.7	180	7.8	180	9.2
240	2.8	240	5.4	240	5.5	240	5.5	240	6.7
300	1.5	300	4.0	300	3.9	300	3.9	300	5.0

(f) Inconel 718

Endurance test, hr					
95		296		500	
Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa	Cycle time, min	Pressure, MPa
13	20.6	16	19.0	15	18.6
47	17.2	55	17.2	37	17.2
60	15.2	60	17.0	60	14.9
120	10.8	120	14.9	120	11.5
180	7.6	180	13.1	180	9.0
240	5.2	240	11.5	240	7.0
300	3.6	300	10.3	300	5.2

TABLE VII. - PERMEABILITY COEFFICIENTS FOR ENDURANCE RUN AT 820° C

Time, hr	Permeability coefficient, $\varphi \times 10^{-6}$ $cm^2/sec\ MPa^{1/2}$					
	Alloy					
	N-155	A-286	Incoloy 800	19-9 DL	HS 188	Inconel 718
5	13.7	21.64	20.6	19.58	11.43	14.7
105	8.39	8.94	8.10	18.70	6.32	4.37
250	6.23	8.06	8.56	17.87	4.95	2.40
500	5.56	----	----	----	4.03	3.45
750	5.27	----	----	----	3.95	----
1000	4.64	----	----	----	3.68	----

TABLE VIII. - TUBING FAILURE TIMES; COMMERCIAL PURITY HYDROGEN, 21 MPa

(a) 860° C endurance run - 535 hr

Alloys	Environment	
	Hydrogen failure time, hr	Helium failure time, hr
N-155	None	489, 534*
A-286	None	261, 277*
Incoloy 800	338, 404, 459	34, 53*
19-9 DL	None	367, 379*, 379*
316 SS	323, 404	53, 63*
Inconel 718	215, 242, 282, 282	275, 323*

(b) 820° C endurance run - 1000 hr

Alloys	Environment	
	Hydrogen failure time, hr	Helium failure time, hr
N-155	None	541, 603*
A-286	405. 556, 895	150, 176*
Incoloy 800	145, 155	31*
19-9 DL	405, 420, 530, 587	359, 389*, 895
HS-188	None	None
Inconel 718	435, 561	256, 266*

*Reduced pressure after this failure.

TABLE IX. - TENSILE PROPERTIES[a] OF TUBING ALLOYS BEFORE AND AFTER ENDURANCE TESTING AT 860° C

(a) Room temperature

Alloy	Environment	Time in rig, hr	Yield strength 0.2 percent offset, MPa	Ultimate tensile strength, MPa	Elongation, percent
N-155	As received	----	480	878	45.0
	Helium	535	376	847	23.5
	Hydrogen	535	400	847	26.5
A-286	As Received	----	212	635	45.7
	Helium	535	303	617	9.0
	Hydrogen	535	312	579	6.0
Incoloy 800	As received	----	181	552	39
	Helium	489	186	557	38.5
	Hydrogen	535	226	554	29.0
19-9 DL	As received	----	290	767	60.3
	Helium	535	300	604	9.0
	Hydrogen	332	311	611	11.5
316 SS	As received	----	293	622	54
	Helium	472	248	603	15.5
	Hydrogen	535	281	618	22.0
Inconel 718	As received	----	404	875	41.5
	Helium	535	466	1060	20.0
	Hydrogen	282	464	965	21.5

TABLE IX. - CONCLUDED

(b) 820 ° C

Alloy	Environment	Time in rig, hr	Yield strength 0.2 percent offset, MPa	Ultimate tensile strength, MPa	Elongation, percent
N-155	As received	----	244	296	24.5[b]
	Helium	535	212	281	42.5
	Hydrogen	535	213	270	45.5
A-286	As received	----	157	199	29.0
	Helium	535	170	216	28.0
	Hydrogen	535	189	220	27.5
Incoloy 800	As received	----	91	162	62.0
	Helium	489	93	156	45.0
	Hydrogen	535	115	143	41.0
19-9 DL	As received	----	147	230	37.5
	Helium	535	135	199	50.0
	Hydrogen	332	151	218	35.0
316 SS	As received	----	119	160	57.0
	Helium	472	122	171	45.0
	Hydrogen	535	122	168	35.0
Inconel 718	As received	----	469	519	19.0
	Helium	535	308	394	31.0
	Hydrogen	282	317	381	28.5

[a]Average of two tests except where noted
[b]Single test

TABLE X. - TENSILE PROPERTIES[a] OF TUBING ALLOYS
BEFORE AND AFTER ENDURANCE TESTING AT 820° C

(a) Room temperature

Alloy	Environment	Time in rig, hr	Yield strength 0.2 percent offset, MPa	Ultimate tensile strength, MPa	Elongation, percent
N-155	As Received	----	480	878	45.0
	Helium	1000	377	834	28.5
	Hydrogen	1000	400	734	8.5
A-286	As Received	----	212	635	45.7
	Helium	1000	234	480	9.8
	Hydrogen	1000	258	592	28.0
Incoloy 800	As Received	----	181	552	39.0
	Helium	1000	218	653	33.0
	Hydrogen	1000	230	538	25.0
19-9 DL	As Received	----	290	767	60.3
	Helium	1000	394	1030	42.5
	Hydrogen	595	366	655	7.0
HS-188	As Received	----	643	1091	35.0
	Helium	1000	538	1125	16.5
	Hydrogen	1000	499	1028	14.5
Inconel 718	As Received	----	404	875	41.5
	Helium	1000	506	1023	22.5
	Hydrogen	1000	481	772	9.0

[a]Average of two tests

TABLE X. - CONCLUDED

(b) 820° C

Alloy	Environment	Time in rig, hr	Yield strength 0.2 percent offset, MPa	Ultimate tensile strength, MPa	Elongation, percent
N-155	As received	--------	244	296	24.5[b]
	Helium	1000	189	279	40.5
	Hydrogen	1000	175	238	24.5
A-286	As received	--------	157	199	29.0
	Helium	895/1000	124	189	25.5
	Hydrogen	1000	177	239	41.5
Incoloy 800	As received	--------	91	162	62.0
	Helium	1000	120	152	49.0
	Hydrogen	1000	120	161	43.0
19-9 DL	As received	--------	147	230	37.5
	Helium	587/595	134	201	27.5
	Hydrogen	925/1000	122	201	29.0
HS-188	As received	--------	359	397	50.0[b]
	Helium	1000	338	392	50.0
	Hydrogen	1000	336	367	40.5
Inconel 718	As received	--------	464	519	19.0[b]
	Helium	1000	299	395	29.0
	Hydrogen	1000	285	361	28.5

[a]Average of two tests except where noted
[b]Single test

TABLE XI. - SUMMARY OF RESULTS

Alloy		External oxidation	Effect of oxygen + CO_2 contained in hydrogen on permeability, ϕ	Tube wall thickness change		Tensile property change, percent of as-received properties							
				860° C 535 hr, percent	820° C 1000 hr, percent	860° C Endurance run				820° C Endurance run			
						Ultimate strength		Elongation, percent		Ultimate strength		Elongation, percent	
						RT	820° C	RT	820° C	RT	820° C	RT	820° C
N-155	He	Thin	---------------	NIL	NIL	-4	-5	-48	+78	-5	-6	-37	+65
	H_2	Thin	Substantially reduced	NIL	NIL	-4	-9	-41	+88	-16	-20	-81	0
A-286	He	Thick	---------------	NIL	NIL	-3	+ 8	-81	-3	-24	-5	-78	-12
	H_2	Thick	Slightly reduced	NIL	NIL	-9	+10	-87	-5	-7	+20	-39	+43
Incoloy 800	He	Thick	---------------	+16	+19	+1	-4	-.01	-27	+18	+6	-15	-21
	H_2	Thick	Slightly reduced	+13	NIL	+.04	-12	-26	-34	-3	+1	-36	-31
19-9 DL	He	Thick (spalled)	---------------	-15	-38	-21	-14	-85	+33	+34	-13	-28	-27
	H_2	Thick (spalled)	Slightly reduced	- 5	-60	-20	-5	-81	-7	-15	-13	-88	-23
316 SS	He	Thick	---------------	NIL	---	-3	+7	-71	-21	---	---	---	---
	H_2	Thick	Substantially reduced	NIL	---	-4	+5	-59	-38	---	---	---	---
HS-188	He	Thin	---------------	---	NIL	---	---	---	---	+3	-1	-53	0
	H_2	Thin	---------------	---	NIL	---	---	---	---	-6	-8	-58	-23
Inconel 718	He	Thin	---------------	NIL	NIL	+22	-24	-52	+63	+17	-24	-46	+53
	H_2	Thin	Substantially reduced	NIL	NIL	+10	-26	-48	+50	-12	-30	-78	+50

TABLE XII. - PREDICTED AND ACTUAL AVERAGE TUBING FAILURE TIMES, 860° C ENDURANCE RUN

Alloy	Temperature, °C	Grain size, μm	Predicted life, hr	Actual life, hr
N-155	862	17	1411	511
A-286	840	73	321	269
Incoloy 800	854	66	32	43
19-9 DL	854	25	512	373
316 SS	871	25	8	58
Inconel	871	26	150	274

Rated/Failure Time in Hours

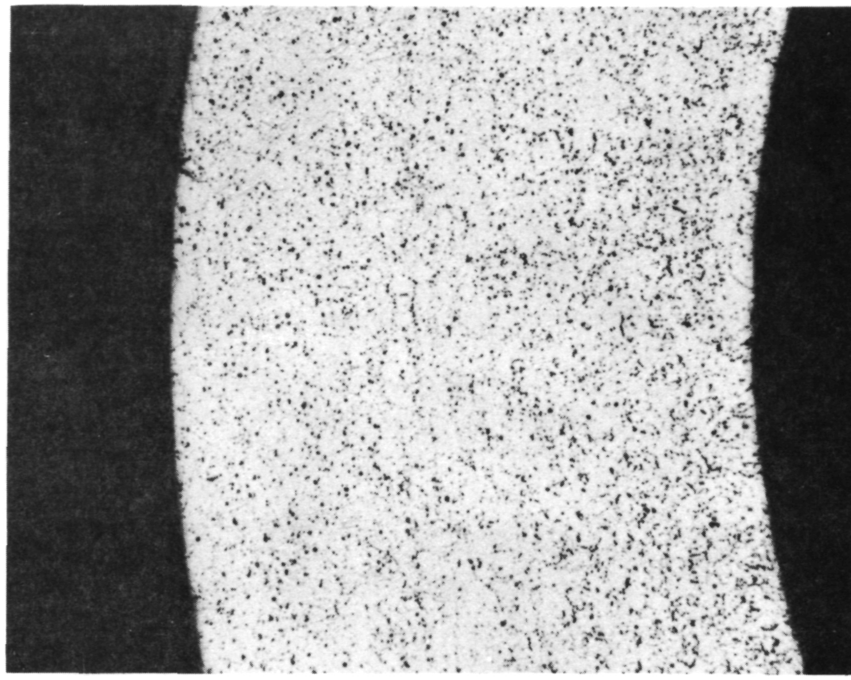
Predicted Rating	Actual Rating
(1) N-155	(1) N-155
(2) 19-9 DL	(2) 19-9 DL
(3) A-286	(3) Inconel 718
(4) Inconel 718	(4) A-286
(5) Incoloy 800	(5) 316 SS
(6) 316 SS	(6) Incoloy 800

TABLE XIII. - PREDICTED AND ACTUAL AVERAGE TUBING FAILURE TIMES 820° C ENDURANCE RUN

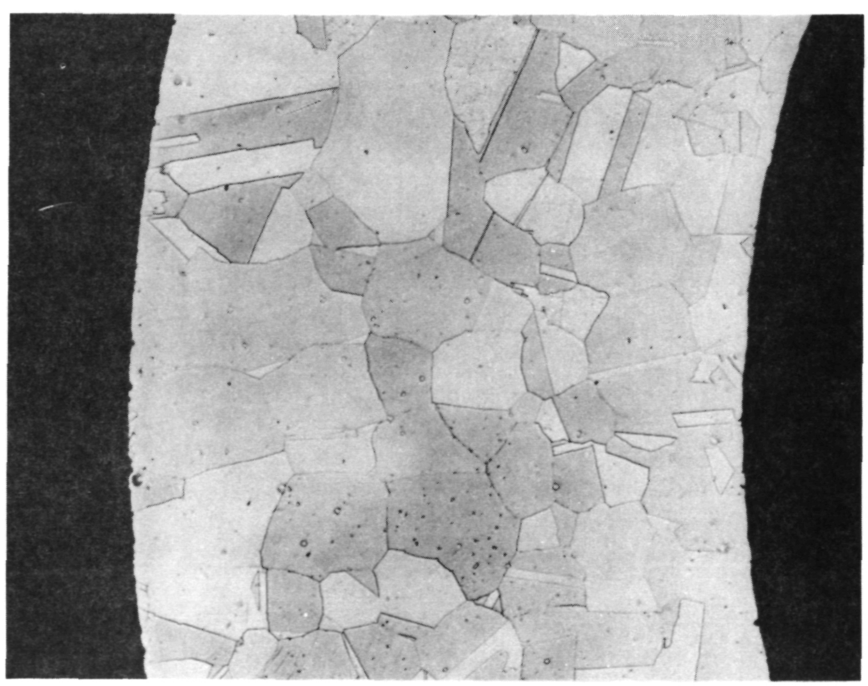
Alloy	Temperature, °C	Grain size, μm	Predicted life, hr	Actual life, hr
N-155	824	17	6 127	572
A-286	821	73	780	163
Incoloy 800	824	66	176	31
19-9 DL	830	25	2 138	375
HS-188	821	7	50 000	No failure
Inconel 718	826	26	1 000	261

Rated/failure time in hours

Predicted rating	Actual rating
(1) HS-188	(1) HS-188
(2) N-155	(2) N-155
(3) 19-9 DL	(3) 19-9 DL
(4) Inconel 718	(4) Inconel 718
(5) A-286	(5) A-286
(6) Incoloy 800	(6) Incoloy 800

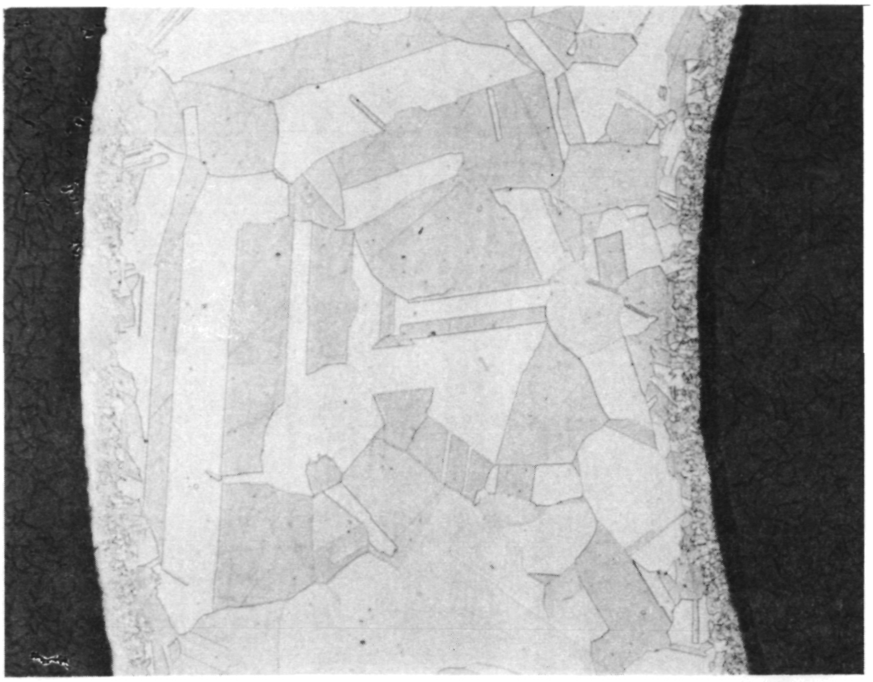


(a) N-155.

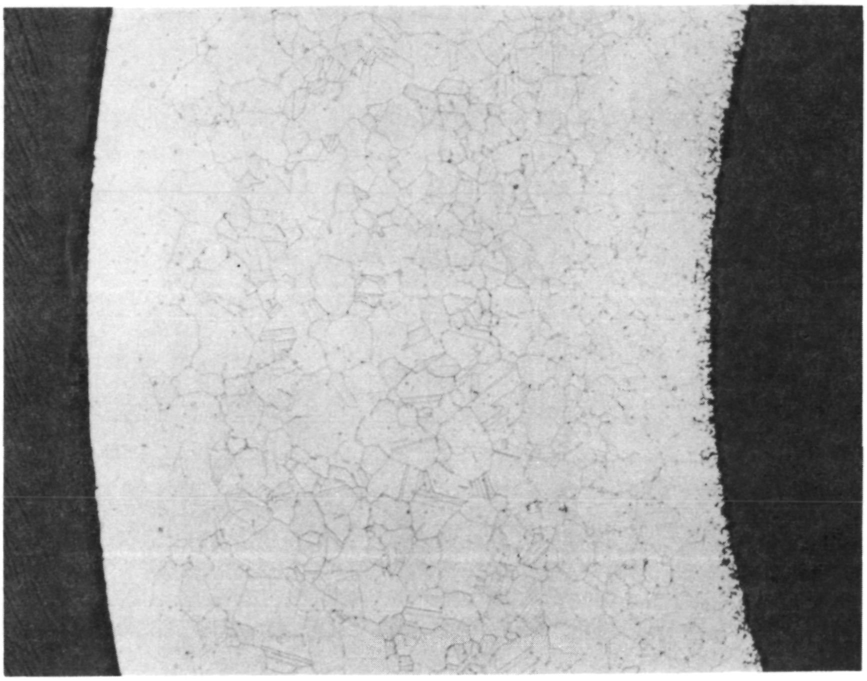


(b) A-286.

Figure 1. - Microstructures of starting materials magnification, 100X.

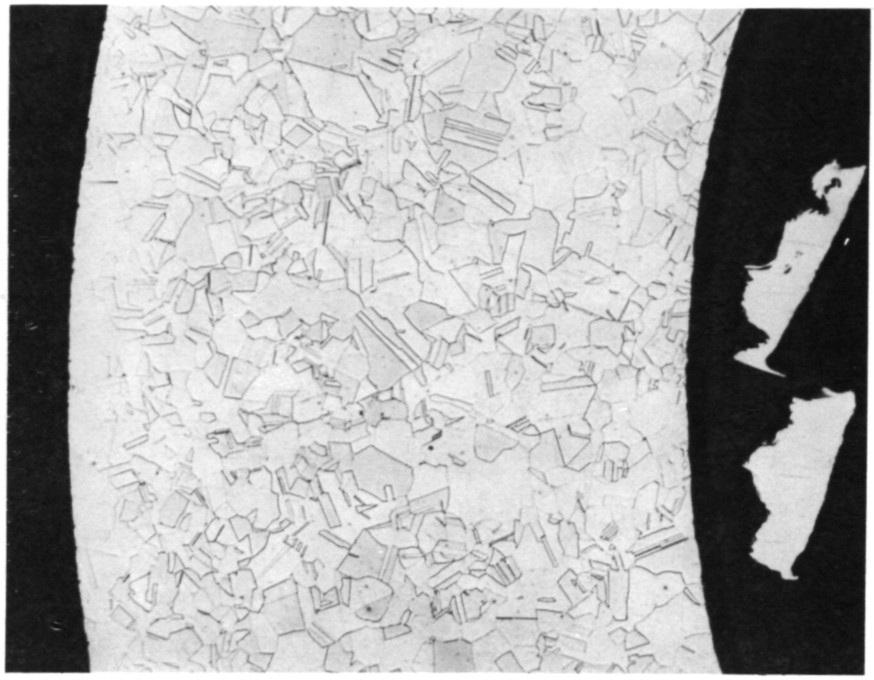


(c) Incoloy 800.

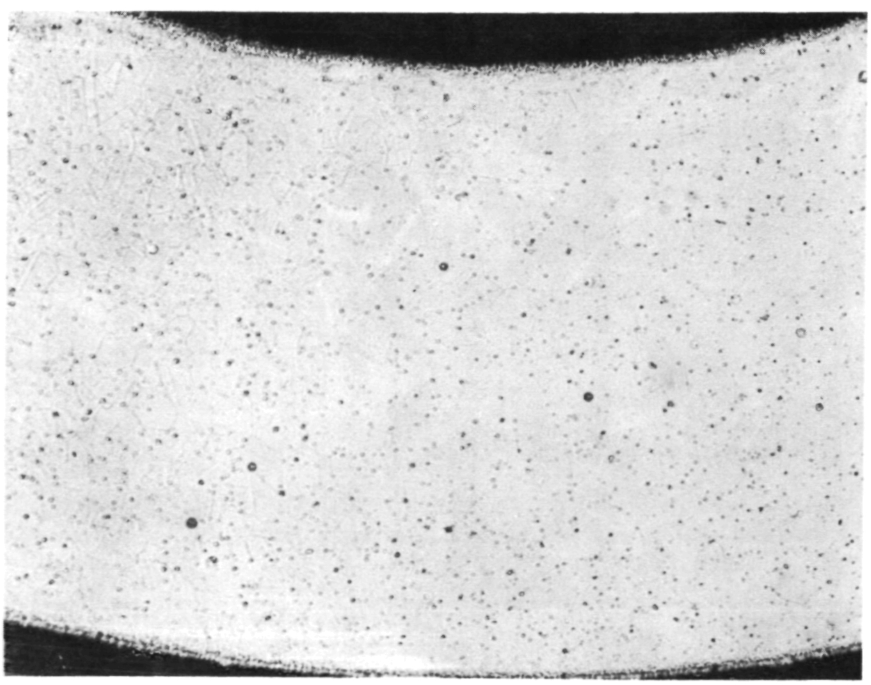


(d) 19-9DL.

Figure 1. - Continued.

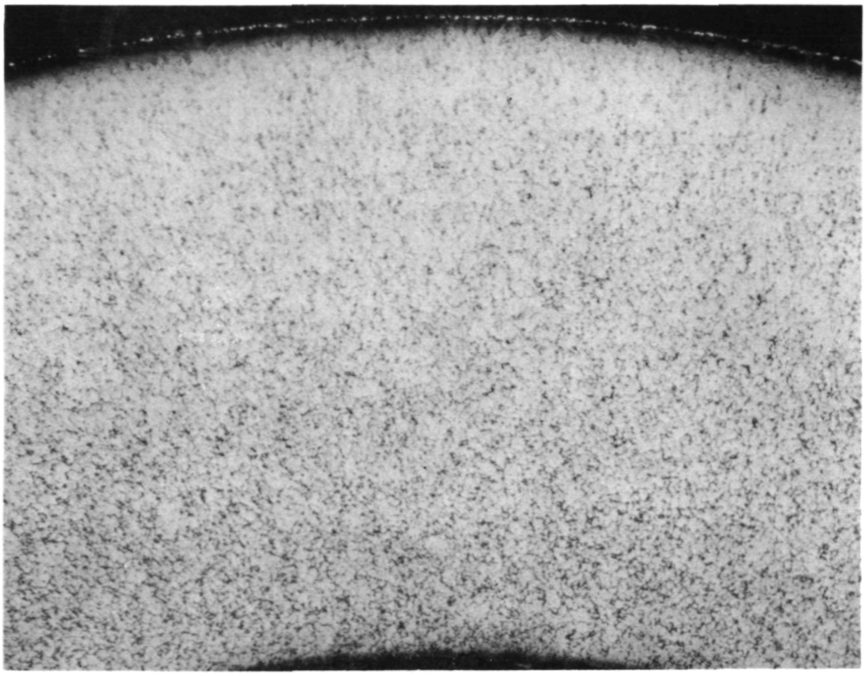


(e) 316 stainless steel.



(f) Inconel 718.

Figure 1. - Continued.



(g) HS-188

Figure 1. - Concluded.

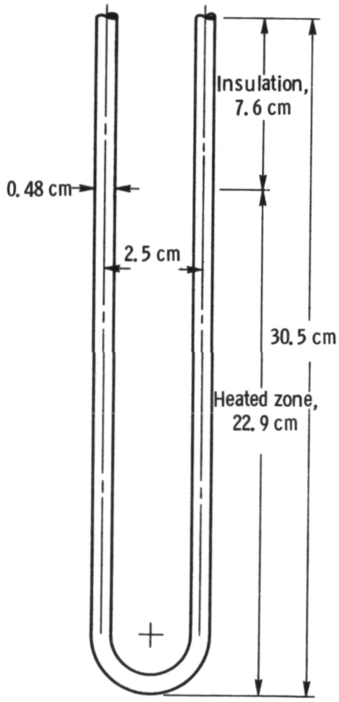


Figure 2. - Hairpin test specimen (not to scale).

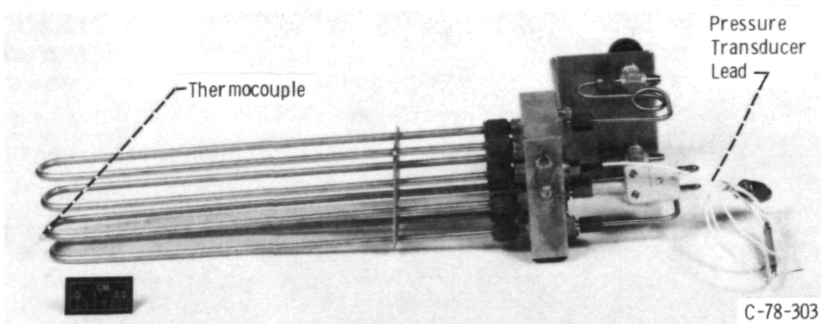


Figure 3. -Test module for Stirling engine simulator materials test rig.

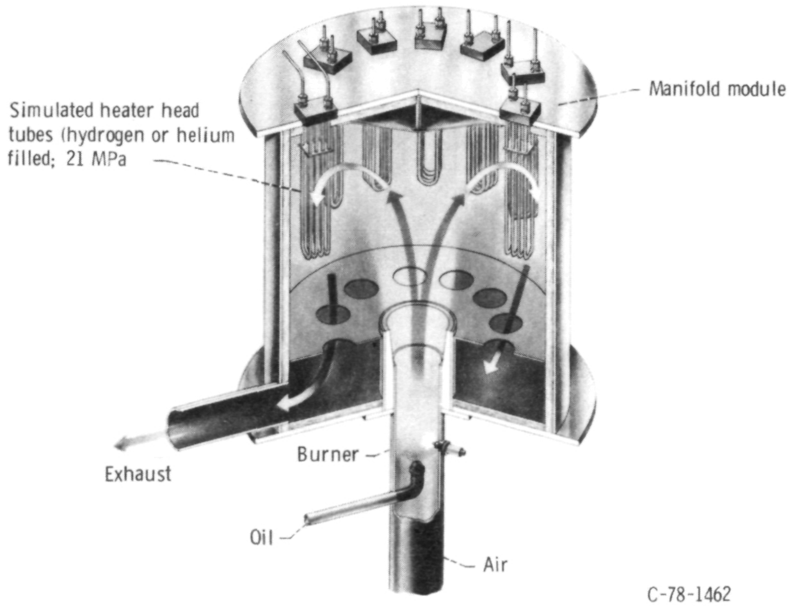
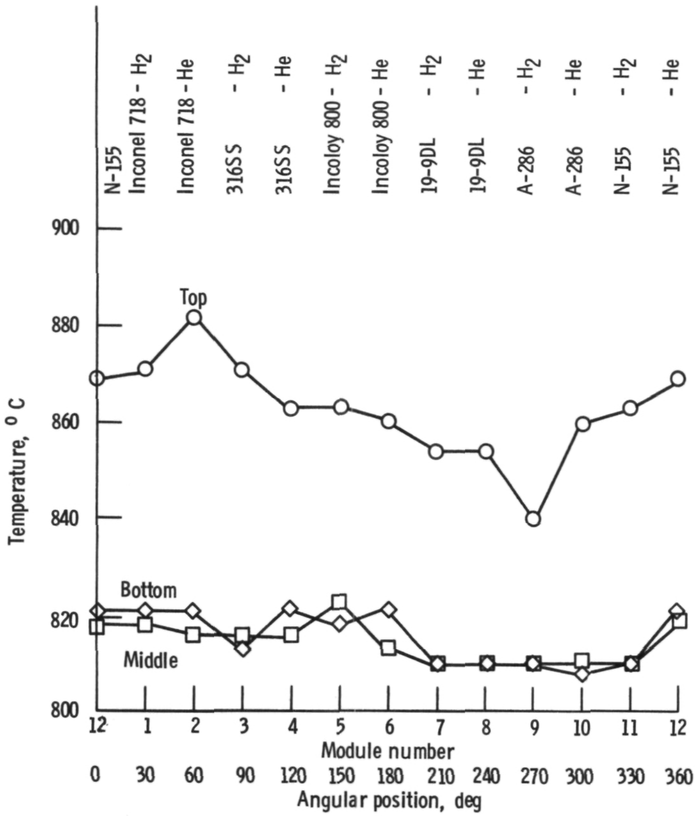
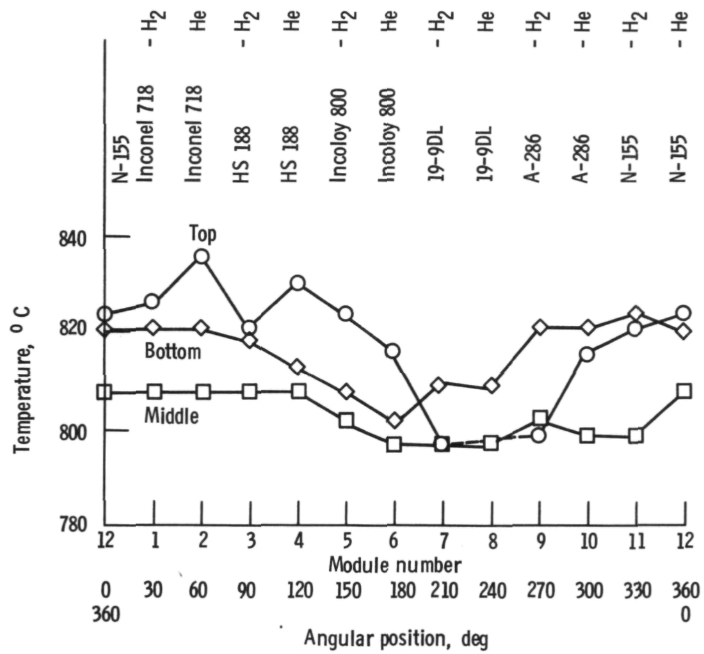


Figure 4. -Schematic representation of Stirling engine simulator materials test rig.



(a) 860° C.

Figure 5. - Temperature profile in Stirling simulator rig for 860° and 820° C endurance runs.



(b) 820° C.

Figure 5. - Concluded.

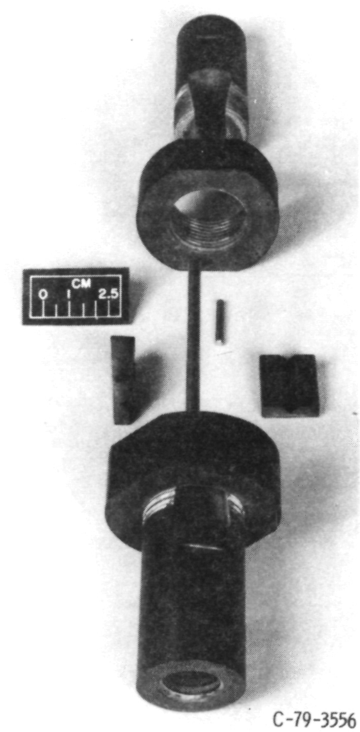
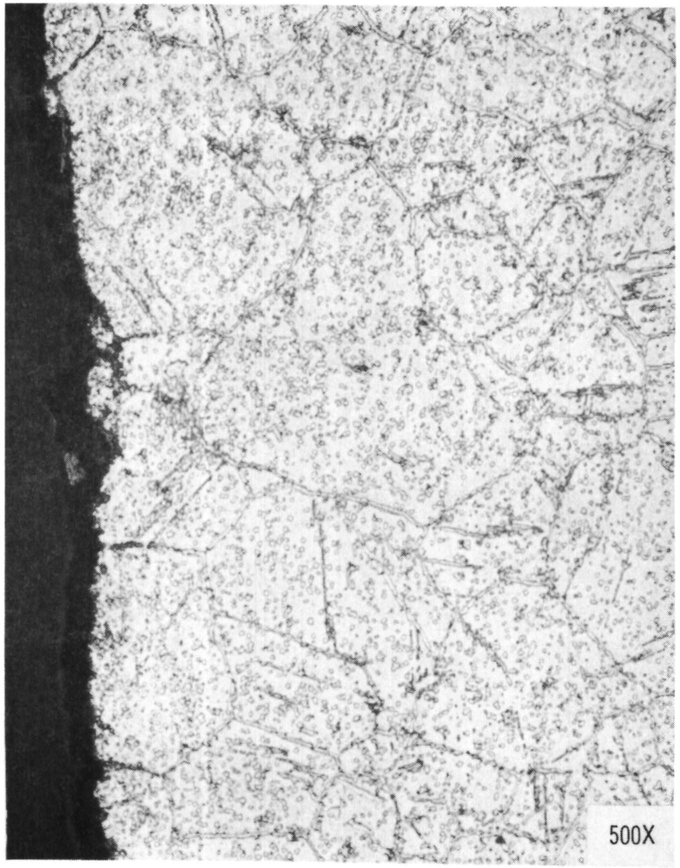
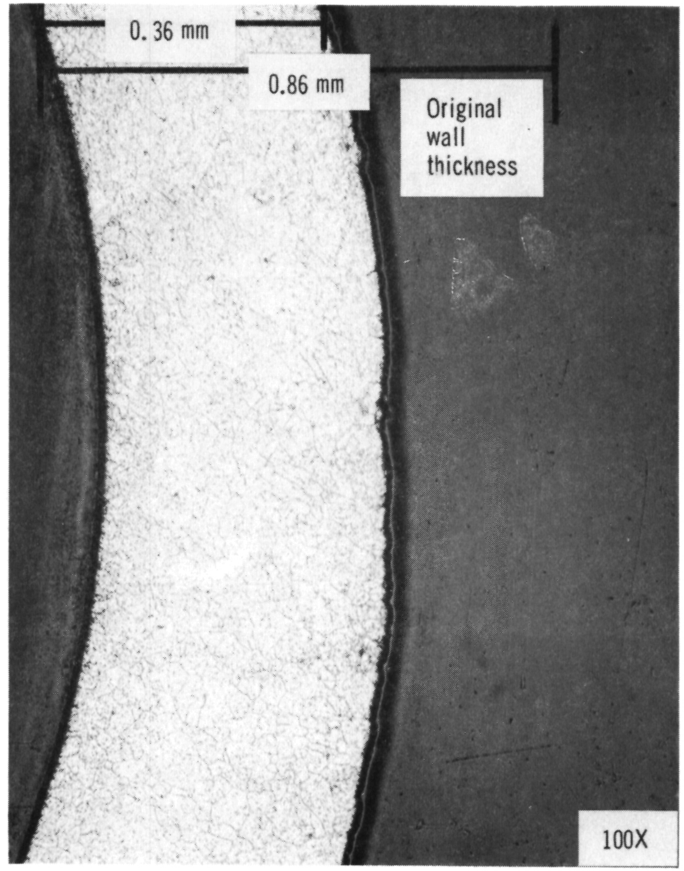


Figure 6. - Tube tensile specimen and grips.

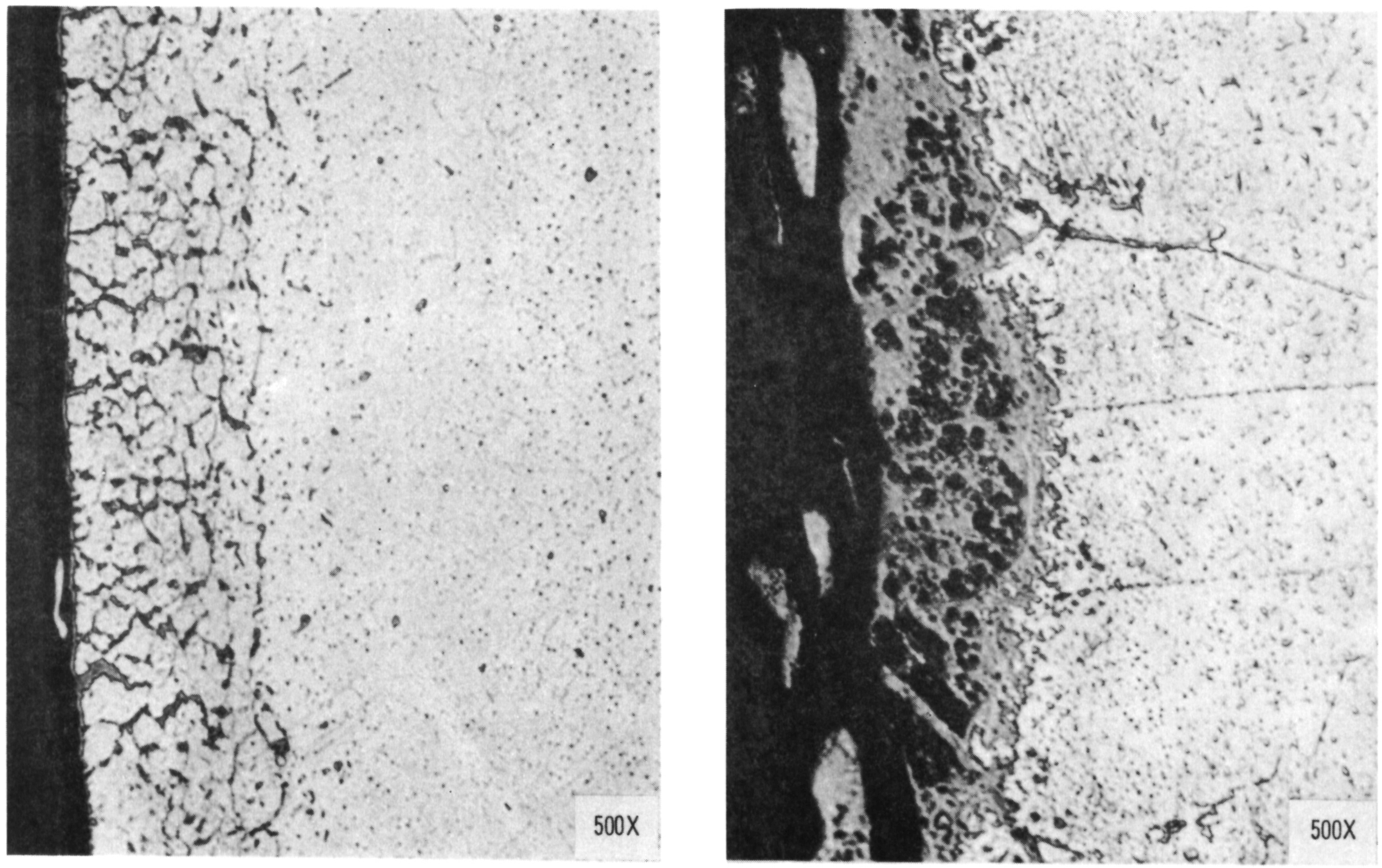


(a) Outside edge of tube.



(b) Tube wall cross section.

Figure 7. - Microstructures of 19-9DL after rig exposure.



(a) Incoloy 800.

(b) A-286.

Figure 8. - Photomicrograph of the outside edge of two alloys after rig exposure.

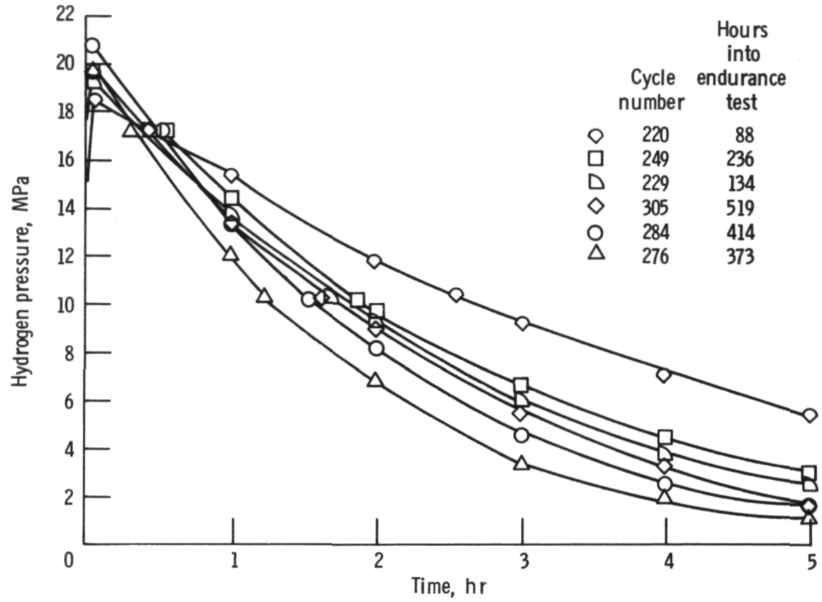


Figure 9. - Typical pressure decay curves for hydrogen filled - Incoloy 800 tubes at 860° C.

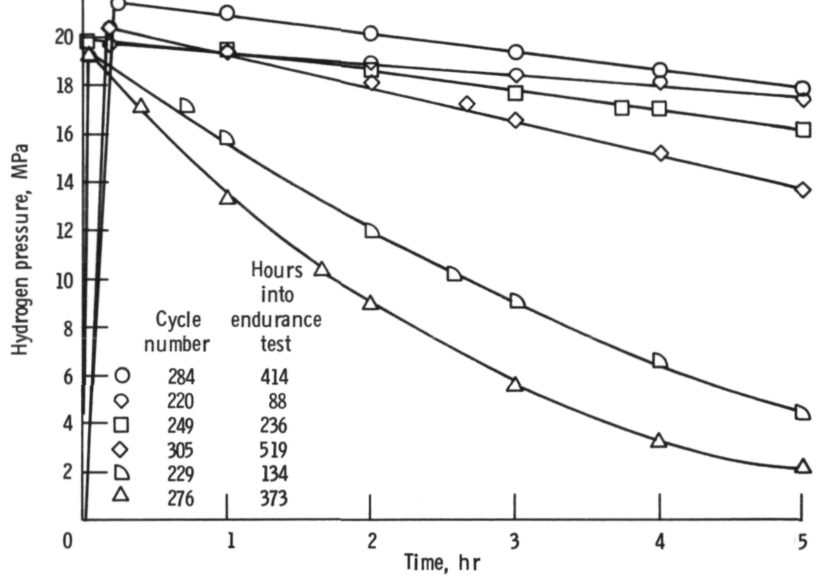


Figure 10. - Typical pressure decay curves for hydrogen filled Inconel 718 tubes at 860° C.

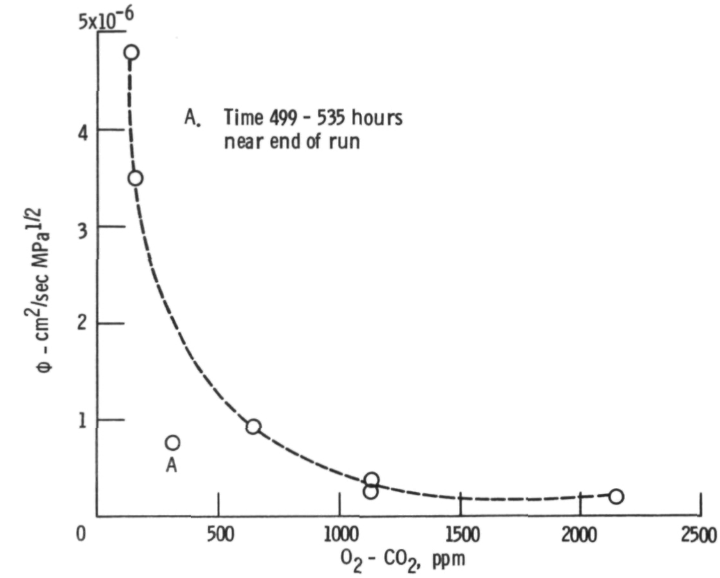


Figure 11. - Effect of O_2 + CO_2 content in commercial purity hydrogen on hydrogen permeability in Inconel 718 at 860° C.

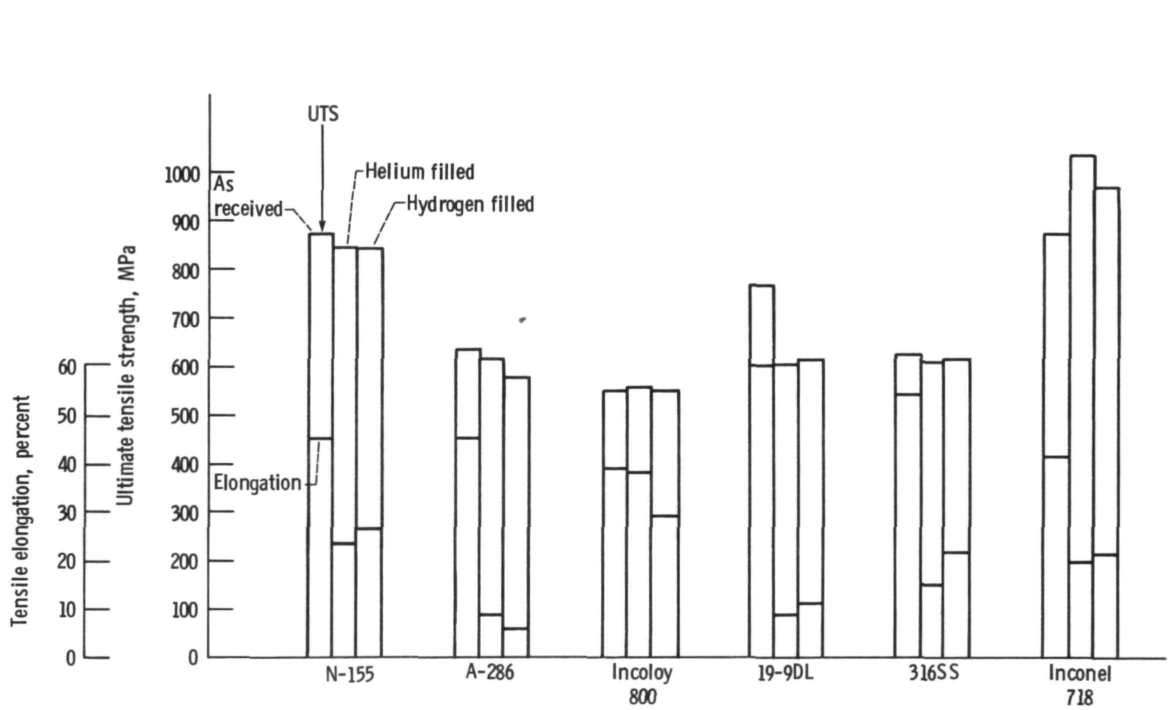


Figure 12. - Room temperature tensile properties of tubing alloys-before and after endurance testing at 860° C - 535 hours.

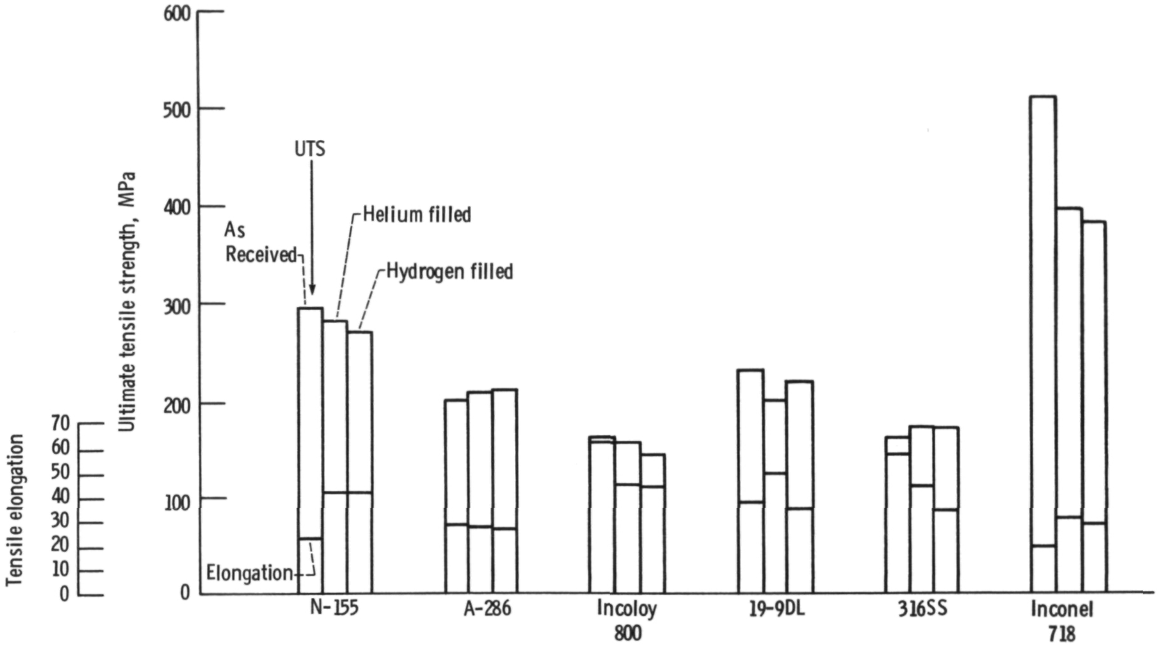


Figure 13. - Tensile properties at 820° C of tubing alloys - before and after endurance testing at 860° C - 535 hours.

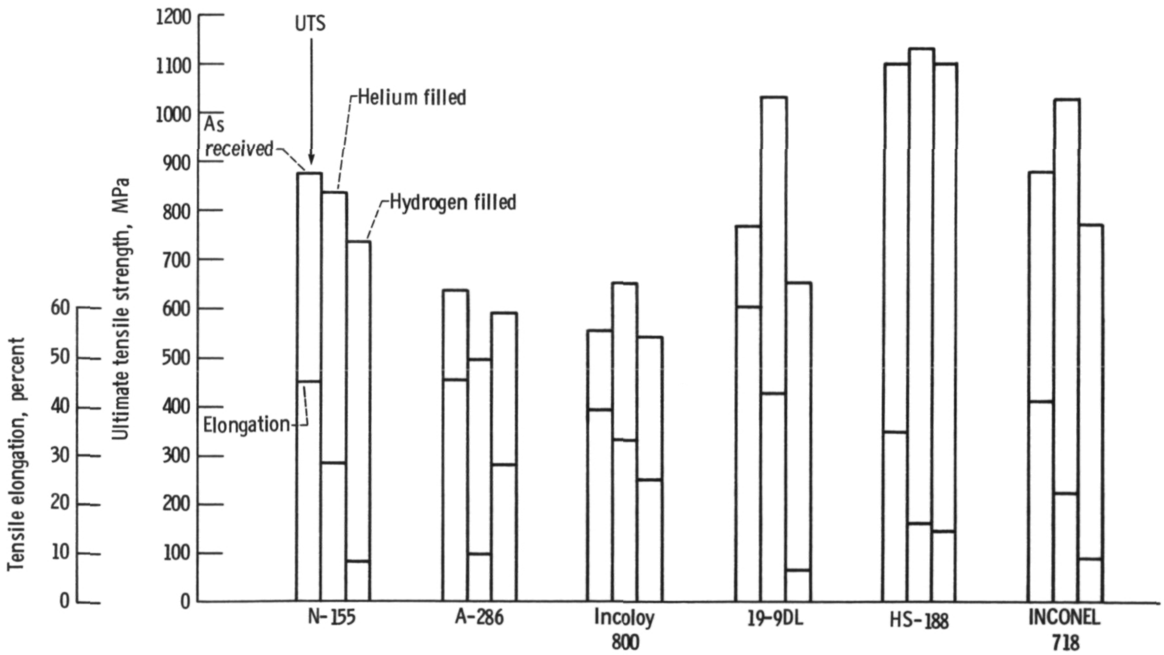


Figure 14. - Room temperature tensile properties of tubing alloys-before and after endurance testing at 820° C - 1000 hours.

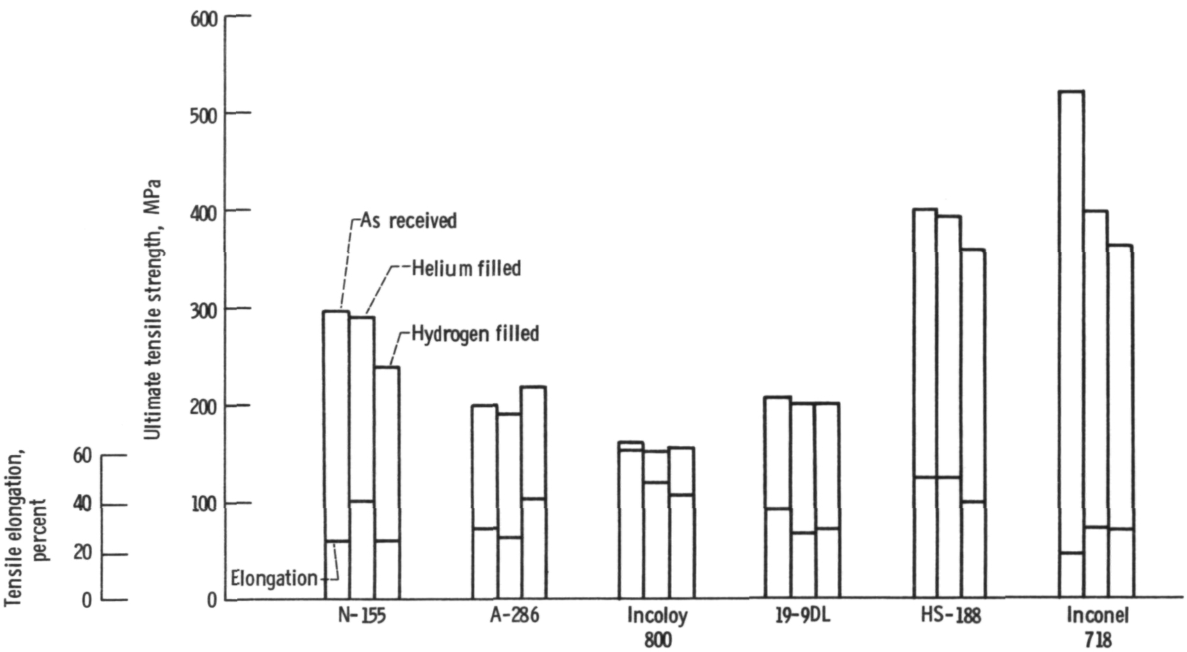


Figure 15. - Tensile properties at 820° C of tubing alloys - before and after endurance testing at 820° C - 1000 hours.

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15. Supplementary Notes

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16. Abstract

Seven commercial alloys were evaluated in the NASA Lewis Research Center Stirling simulator materials rigs. Five iron-base alloys (N-155, A-286, Incoloy 800, 19-9DL, and 316 stainless steel), one nickel-base alloy (Inconel 718), and one cobalt-base alloy (HS-188) were tested in the form of thin-wall tubing in a diesel-fuel-fired test rig. Tubes filled with hydrogen or helium at gas pressure of 21.6 MPa and temperatures of 820° and 860° C were endurance tested for 1000 and 535 hours, respectively. Results showed that under these conditions hydrogen permeated rapidly through the tube walls, thus requiring refilling during each 5-hour cycle. Helium was readily contained, exhibiting no measurable loss by permeation. Helium-filled tubes tested at 860° C all exhibited creep-rupture failures within the 535-hour endurance test. Subsequent tensile test evaluation after removal from the rig indicated reduced room temperature ductility for some hydrogen-filled tubes compared to helium-filled tubes, suggesting possible hydrogen embrittlement in these alloys.

17. Key Words (Suggested by Author(s)) Stirling engine; Materials; Hydrogen; Permeability; Endurance tests 820° and 860° C	18. Distribution Statement Unclassified - unlimited STAR Category 26 DOE Category UC-25

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